DEVELOPMENT OF VARIOUS SALTS ASSORTED PEO BASED BLEND POLYMER ELECTROLYTE FOR SUPERCAPACITOR APPLICATIONS

SYNOPSIS

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1. INTRODUCTION

Modernization of the world demands energy storage devices to be lighter, safer, flexible and cost effective and hence researchers are motivated to develop more accommodating designs for future applications. The energy devices have two important components i) electrolyte and ii) electrodes and each of them plays an important role in the device.

Solid State Ionics (SSI) is an interdisciplinary area of Science and Technology that deals with the study of ionic solid having/exhibiting a wide range of ionic conductivity from 10^{-13} – 10^{-1} S cm^{-1}. The class of Solid Material having ionic conductivity of the order of 10^{-5} – 10^{-1} S cm^{-1} comparable to those liquid electrolytes are referred to as "Fast Ion Conductor" or "Superionic Solid". These materials (show tremendous promises) have a wide variety of applications in electrochemical devices such as batteries, supercapacitors, fuel cell sensors, electrochromic display devices, etc. [1].

A Superionic Solid or Solid Electrolytes have the following properties:

- High ionic conductivity of the order of 10^{-5} – 10^{-1} S/cm.
- Ions as principle charge carriers i.e., $t_{ion} \approx 1$.
- Negligible electronic conductivity (Small electronic transference number ($t_{ele} < 10^{-4}$)
- Low Activation Energy (0.1 eV – 1 eV).
- Polymer electrolytes should possess the following properties to be used in the development of solid-state devices:
- High ionic conductivity $\sigma \geq 10^{-4}$ S cm^{-1} at room temperature close to liquid electrolytes.
- High ionic transference number t_{ion} ~1.
- Good mechanical, thermal, and chemical stability. High chemical stability is required to avoid chemical reaction at electrode-electrolyte interfaces.

- Wider electrochemical stability window as high as 4 to 5 V.
- Low activation energies for ion conduction.

In this respect solid polymer electrolytes are an alternate option because of having soft and flexible physique which can be molded in desired shape and size etc. supporting the designing ease. Polymer electrolytes can be classified in different categories such as i) gel polymer electrolyte, ii) composite polymer electrolyte, iii) plasticized polymer electrolytes, iv) polymer-salt complex/solvent free electrolytes, etc. In polymer-based electrolytes, polymer salt complexes are the most studied one, because of ease in their fabrication and molding them in different shapes and sizes.

The present thesis deals with polymer-salt electrolyte. Blending of polymers is an easy and useful method in developing polymeric materials with improved mechanical stability and with desirable properties. The blending of two or more commercially available polymers in order to obtain new products of desirable properties is one of the simplest and widely applied concepts in the field of materials science. Utilization of polymer electrolytes in the electrochemical devices' challenges with optimization of ionic conductors with desirable properties along with the knowledge of ion transport mechanism in them.

Our research focus is mainly on PEO, as primary host polymer, PVP and P(VdF-HFP) has been chosen as copolymer respectively. The present study deals with the effect of Ammonium salts of different concentration in PEO/PVP matrix, and Ammonium, lithium, sodium and magnesium salts of different concentrations in the PEO/P(VdF-HFP) matrix with an aim to obtain high ionic conductivity than pure blend systems.

The present solid polymer electrolyte system is comprised of poly ethylene oxide (PEO), poly (vinylidene fluoride hexafluoropropylene) P(VdF-HFP) and poly vinylidene pyrrolidine as host polymer. PEO has received a lot of attention due to semi-crystalline polymer, possessing both amorphous and crystalline phases at room temperature. As PEO is a linear polymer, the regularity of the structure unit allows a high degree of crystallinity. The chemical structure of PEO consists of the sequential oxy-ethylene group $–CH_2–CH_2–O–$ that has series of polar group –O– which can associate with metal cations. Hence PEO can solvate a wide variety of salts even at very high salt concentrations. However, PEO itself only has C–C, C–O, C–H bonds, the

reactivity is exceedingly low and because of this, it is stabilized chemically and electro chemically [2]. Unfortunately, a high crystalline phase concentration limits the conductivity of PEO based electrolytes; this is due mainly to the basic requirement for ionic mobility, i.e., ionic motions coupled to the segmental relaxations of a flexible amorphous polymer phase.

Along with this P(VdF-HFP) is blended with PEO owing to its excellent properties such as a low degree of crystallinity and high dielectric constant $\varepsilon \approx 8.4$ that help in higher dissociation of salt. The P(VdF-HFP) semicrystalline polymer possesses both amorphous and crystalline phases in which the amorphous HFP part can trap large amounts of liquid electrolytes whereas the crystalline VDF part assists in the improvement of the mechanical stability for the formation of a free-standing film [3]. Along with this P(VdF-HFP) is used as a blender with PEO polymer due to its advantageous properties such as good compatible nature with other polymers, high chemical resistance, electrical properties, and highly amorphous in nature which aid for increasing the amorphicity [4].

Polyvinyl pyrrolidone (PVP) was selected as the second polymer component for preparing polymer blend with PEO, because of its unique characteristics. First PVP is an amorphous polymer, which can permit faster ionic mobility compared to other semicrystalline polymers and has low scattering loss, which makes it an ideal polymer for blend-based materials for optical applications. Second, due to the presence of carbonyl group (C = O) in the side chains of PVP, it forms a variety of complexes with various inorganic salts. It interacts well with several types of ions and enhances the number of free ions in the system. Another advantage of using PVP is that it can be thermally crosslinked, resulting in outstanding thermal stability and mechanical strength of the blend material. Furthermore, PVP is highly soluble in polar solvents such as alcohol [5].

LITERATURE SURVEY

Jayanthi [6] has reported that the PEO/P(VdF-HFP) as polymer hosts, zinc triflate (ZnTr) as an electrolyte, and 1-butyl-3-methylimidazolium hexafluorophosphate (1B3MIHFP) as an ionic liquid (IL) via a conventional solvent casting technique. From AC impedance spectroscopic studies, maximum ionic conductivity of 9.572×10^{-4} S cm^{-1} was observed for the system with 20 wt.% of 1B3MIHFP at room temperature. Jayanthi [7] has found that the PEO/P(VdF-HFP)

polymer blend, it was complexed with lithium perchlorate ($LiClO_4$), and nanosized strontium titanate (70.60 nm) $SrTiO_3$ particles were dispersed into the complex in different weight ratios. The maximum ionic conductivity, 4.827×10^{-5} S cm^{-1}, was obtained for 2 wt.% $SrTiO_3$. Jayanthi [8] observed that the Nanocomposite polymer blend electrolytes composed of PEO: P(VdF-HFP): $LiClO_4$ were prepared for various concentrations of TiO_2 nanofiller. In addition, filler concentration in the complex was also optimized based on ionic conductivity, and it was noted that the sample containing 6 wt.% showed maximum value about 1.451×10^{-5} S cm^{-1} at room temperature. In pradeepa prabakaran [9] has reported that Polymer blend electrolytes based on poly(ethylene oxide) (PEO) and poly(vinylidene fluoride hexafluoropropylene) P(VdF-HFP) were prepared by using different lithium salts Li X (X = ClO_4, BF_4, CF_3SO_3, and N $[CF_3SO_2]_2$) using solution casting technique. AC impedance analysis was performed for all the electrolyte samples at various temperatures from 303 to 343 K. The result suggests that among the various lithium salts, Li $N[CF_3SO_2]_2$-based electrolytes exhibited the highest ionic conductivity at 8.20×10^{-4} S/cm. In Sundaramahalingam [10] has reported that the polyethylene oxide (PEO) /Polyvinyl pyrrolidone (PVP) based Solid polymer electrolytes (SPEs) was prepared with lithium bromide by solution casting technique. The maximum ionic conductivity was found to be 1.59×10^{-6} S cm^{-1} for PEO (30wt.%) /PVP (70wt.%) /Lithium bromide (4wt.%) at 303K within the frequency range of 42 Hz – 1 MHz. Sundaramahalingam [11] has reported that solid polymer electrolytes (SPEs) consisting of polyethylene oxide (PEO) /Polyvinyl pyrrolidone (PVP) complexed with sodium nitrate have been synthesized by solution casting technique. The ionic conductivity and dielectric response of the SPE systems were studied within the frequency range of 42 Hz - 1 MHz at the temperature range of 303-363 K. The maximum ionic conductivity was found to be 6.157×10^{-7} S/cm at 303K for PEO (67wt.%) /PVP (27wt.%)/$NaNO_3$ (6wt.%). Sharanappa Chapi [12] has reports the polyethylene oxide/ polyvinylpyrrolidone (PEO/PVP) blend with cobalt chloride ($CoCl_2$) films prepared using spin coating method on blue star glass substrate. The maximum ionic conductivity 0.65×10^{-4} S cm^{-1} was observed for PEO (45%)/PVP (45%)/$CoCl_2$ (10 %) at 30 °C.

OBJECTIVES

The need for environment friendly energy storage systems, non-explosive is a big driving force for materials researchers to develop polymeric materials which have

the properties to maintain the highest level of energy in an energy storage system. With this big goal in mind, the researcher emphasizes on contributing to the fascinating research area of polymer electrolytes. The following are the main objectives of the present research

- To synthesize free-standing solid blend polymer electrolyte films based different ion (Li^+, Na^+, Mg^{2+}, NH_4^+) based different blend (PEO/PVP, PEO/P(VdF-HFP)) polymer electrolyte using solution casting technique.
- To obtain an optimum polymer-salt composition for maximum ionic conductivity for the solid blend polymer electrolyte.
- To investigate the effect of some different salt on the structural, vibrational and electrical properties of the polymer electrolyte systems with the perspective of electrochemical device application.
- To test the ability of the prepared ionically conducting polymer electrolytes in the fabricated EDLCs.

RESEARCH PROBLEM

The primary criteria of electrolytes for energy storage device applications are i) high ionic mobility, ii) high electrochemical stability, iii) environmentally friendly nature, iv) adaptability and v) excellent electrode compatibility nature. By mixing i) inorganic salt, ii) plasticizer, iii) ionic liquid and iv) filler with SPEs, this issue may be solved. In this dissertation, various inorganic salts are used to enhance ion conduction in SPEs. Blending polymers can help create novel polymeric materials with enhanced mechanical stability and electrical conductivity. The blend system's key benefits are its ease of preparation and ability to easily adjust physical qualities through compositional change. Solid polymer electrolyte materials have attracted the attention of researchers in recent years due to their potential uses in ionic gadgets including i) rechargeable batteries, ii) supercapacitors, iii) gas sensors, iv) fuel cells, v) nano architectonics, vi) electrochromic display displays/smart windows, and so on.

With the view of the research on solid blend polymer electrolytes, the following research pattern was achieved for the work undergone and is presented in figure 1.

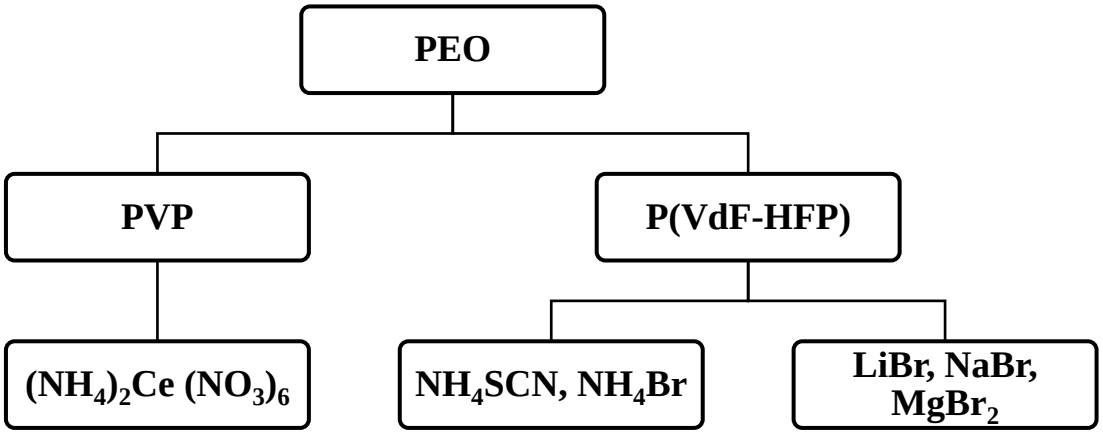

Figure 1: Schematic illustration of the research pattern of the work undergone.

ORGANIZATION OF THESIS

The entire work of the thesis is organized in **Nine** chapters as summarized below.

Chapter I: *Introduction"* mainly consists of general introduction, basic aspects, types of polymer electrolytes and their applications**.**

Chapter II: *"Literature survey"* it contains a brief review of literature. An outline of the scope of the present investigation is provided towards the end of this chapter.

Chapter III: *"Materials and methods"* describes material properties, experimental aspects, different methods of preparation of polymer complexes, and characterization studies such as XRD, FT-IR, AC impedance spectroscopy, Transference number, LSV, CV etc. are explained. The details of the experimental techniques used for the preparation and characterization of polymer electrolyte materials have been described. The necessary background and detailed description of the physical techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), are given for structural ion conducting polymers. Impedance spectroscopy for ionic conductivity measurements, transport number measurement techniques and cyclic voltammetry are presented for electrochemical characterization of the electrolytes. The electrochemical preparation techniques for the preparation of conducting polymer are discussed. Finally, the fabrication of solid-state supercapacitors and their characterization using cyclic voltammetry (CV) are presented.

Chapter VI: *"Ionic conductivity and dielectric studies on PVP/PEO/ $(NH_4)_2$ Ce $(NO_3)_6$ based solid polymer-blend electrolytes"* deals with, proton ion-conducting PVP (poly vinylidene pyrrolidine), PEO (poly ethylene oxide), blend-based polymer electrolytes with various compositions of $(NH_4)_2Ce\ (NO_3)_6$ salt are being prepared and characterized. With increasing compositions of $(NH_4)_2Ce\ (NO_3)_6$, the ionic conductivity of the polymer-blend electrolytes is increasing. The salt-containing 1.5 wt.% system shows higher ionic conductivity of 2.88×10^{-5} S cm^{-1} at room temperature. The ionic conductivity of the polymer electrolytes obeys Arrhenius's behaviour. Dielectric properties of the prepared electrolytes are also analyzed. From Wagner's polarization technique, it is confirmed that the conduction in the polymer electrolytes is predominately due to ions.

Chapter V: "*Preparation and characterization of proton (H+) conducting solid blend polymer electrolytes based on PEO/P(VdF-HFP) incorporated with NH_4SCN" and "Fabrication and characterization of NH_4^+ – ion-conducting flexible solid blend polymer electrolytes based on poly (ethylene oxide) and poly (vinylidene fluoride-co hexafluoropropylene)"* deals with the preparation and characterization of PEO/P(VdF-HFP)/ different wt.% NH_4SCN system and NH_4Br system respectively. The comparison of structural and vibrational data of both systems were presented. The ionic conductivity has been found to be high for PEO/P(VdF-HFP)/20 wt.% NH_4SCN and PEO/P(VdF-HFP)/20 wt.% NH_4Br electrolyte films and has been found to be 2.69×10^{-5} and 2.55×10^{-4} S cm^{-1} at 303 K, respectively. Based on the free-standing of the film at higher wt.% and ionic conductivity, PEO/P(VdF-HFP)/different wt.% NH_4Br system shows to be the best solid polymer blend conducting electrolyte. Hence, the optimized samples of PEO/P(VdF-HFP)/NH_4Br can be utilized for supercapacitor applications.

C**hapter VI:** "*Characterization of lithium-based poly (ethylene oxide)/poly (vinylidene fluoride-co-hexafluoropropylene) solid blend polymer electrolytes for energy storage applications"* presents poly (ethylene oxide) (PEO): poly (vinylidene fluoride-co hexafluoropropylene) P(VdF-HFP): lithium bromide (LiBr)-based solid blend polymer electrolytes (SPEs) are prepared by using solution casting technique with dimethyl formamide (DMF) as solvent. According to XRD analysis, the incorporation of LiBr improves the amorphous nature of the prepared electrolytes. FTIR analysis confirms the complexation between polymers and salt. Scanning electron microscopy (SEM) was used to examine the surface morphology and blending of the

electrolytes. Impedance spectroscopy studies showed higher ionic conductivity of 3.75 × 10^{-4} S cm^{-1} at room temperature for the sample containing 4 wt.% LiBr. The electrolytes were observed to follow Arrhenius behavior with $R^2 \sim 1$, in which all samples were thermally activated as the temperature rises. The complex dielectric permittivity (ε^*), loss tangent ($tan\ \delta$), conductivity (σ), and also complex electric modulus ($M*$) were used to understand the presence of electrode polarization. Wagner's polarization technique was used to determine the transference number of these polymer electrolytes and it was found that the ionic conductivity was mostly due to ions. Transport characteristics like mobility (μ) and mobile ion diffusion coefficient (D) were also derived. The breakdown voltage of the polymer electrolytes was evaluated using linear sweep voltammetry (LSV). Using cyclic voltammetry (CV) analysis as well as Galvanostatic charge–discharge (GCD) measurement, maximum specific capacitance (*Csp*) of 11.27 F/g was determined for the electrolytes. The energy (E) and power (P) density have been found to be 1.34 Wh/kg and 250 W/kg respectively.

Chapter VII: "*Enhancement of electrical and electrochemical properties of sodium bromide incorporated with poly (ethylene oxide)/poly (vinylidene fluoride hexafluoropropylene) solid blend polymer electrolytes for electrochemical double layer capacitors"* describes PEO/P(VdF-HFP) system with the incorporation of different wt.% of NaBr salts. Sodium-ion conducting solid blend polymer electrolytes are prepared with Poly (ethylene oxide) (PEO)/Poly (vinylidene fluoride-hexafluoropropylene) P(VdF-HFP)/Sodium Bromide (NaBr) by solution casting technique and is shown in figure 2. Different analysis such as structural, vibrational, electrical and electrochemical studies reveal promising properties of the prepared solid polymer electrolytes (SPE). The XRD analysis illustrates the increase in amorphous nature of the sample up to 8 wt.% NaBr salt concentration. The complexation between polymers and salt has been examined by FTIR analysis. To examine the electrical conductivity, AC impedance analysis was performed in the temperature range of 303–373 K. The higher ionic conductivity (σ) of 1.10 × 10^{-3} S cm^{-1} was found for the solid polymer electrolyte (SPE) containing 8 wt.% NaBr at room temperature. The temperature dependence of ionic conductivity resembles Arrhenius behavior. The dielectric behavior has been analyzed using dielectric permittivity (ε^*) and the relaxation frequency (τ) has been calculated from the loss tangent spectra ($\tan \delta$). The modulus spectra indicate the non-Debye nature of the material. Wagner's DC polarization technique was used to compute the transference number. According to the

LSV analysis, the solid blend polymer electrolytes have the suitable electrochemical potential window to be used in electrochemical devices. The cyclic voltammetry (CV) profile and the charge-discharge profile (GCD) of the assembled electrochemical double-layer capacitor (EDLC) as per the schematic illustration in figure 3, revealed specific capacitance (C_{sp}) values of 12.9 F/g and 10.9 F/g, respectively. Further, other significant aspects for assessing EDLC performance, including energy density (1.51 to 0.93 Wh/kg), power density (125 to 1250 W/kg), as well as coulombic efficiency (97.2%) after 500 cycles, have been determined.

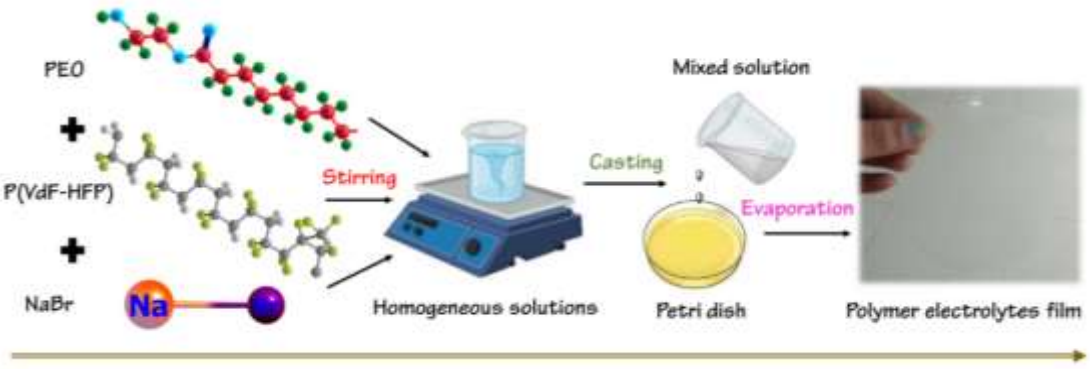

Figure 2: Schematic illustration of the Preparation methods of Polymer electrolytes

Figure 3: Schematic illustration of the Electrode fabrication.

Chapter VIII: "*Electrical properties of Mg^{2+} ion-conducting PEO: P(VdF-HFP) based solid blend polymer electrolytes"* the effect of addition of $MgBr_2$ in PEO/P(VdF-HFP) polymer electrolyte system is explained. Mg^{2+} ion-conducting solid blend polymer electrolytes (SPEs) based on PEO: P(VdF-HFP) are prepared by solution casting technique using DMF as solvent. The X-ray diffraction analysis (XRD) confirms the decrease in crystalline phase of polymer electrolytes due to the addition of salt. The complex formation between polymers and salt is confirmed by Fourier transform infrared spectroscopy (FTIR) analysis. Morphological analysis shows the

existence of interconnected micro-pores that enhances the mobility of Mg^{2+} ions. The polymer electrolyte (PEO: P(VdF-HFP): $MgBr_2$) (90:10:15) has higher ionic conductivity of 3.90×10^{-4} S/cm at room temperature. For all solid blend polymer electrolytes, the total ionic transference number has been observed to be 0.98–0.99 range. The polymer electrolytes exhibit maximum electrochemical stability of 1.86 V. The cyclic voltammetry (CV) performance of the prepared EDLC reveals the increase in specific capacitance as the scan rate decreases. According to charge-discharge studies, the cyclic retention of the EDLC is maintained to be ~84.2% even after 500 cycles.

Finally, the summary of the findings on the topic of investigation in a proper perspective is given in **chapter IX**. Among the all systems, NaBr doped solid polymer blend electrolyte show the highest ionic conductivity. 90 wt.% PEO: 10 wt.% P(VdF-HFP): 8 wt.% NaBr is observed to have maximum conductivity 1.10×10^{-3} S cm^{-1} at room temperature. The cyclic voltammetry (CV) profile and the charge-discharge profile (GCD) of the assembled electrochemical double-layer capacitor (EDLC) revealed specific capacitance (C_{sp}) values of 12.9 F/g and 10.9 F/g, has been observed. Further scope to synthesize a new, highly conducting solid polymer electrolyte is also elucidated.

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LISTS OF PUBLICATION

1. **S. Shenbagavalli,** M. Muthuvinayagam, M. S. Revathy, & P. Sasikumar, Ionic conductivity and dielectric studies on PVP/PEO/$(NH_4)_2Ce(NO_3)_6$ based solid polymer-blend electrolytes. *Bulletin of Materials Science*, **45** (2022) 125. https://doi.org/10.1007/s12034-022-02713-5.

2. **S. Shenbagavalli,** M. Muthuvinayagam, & M. S. Revathy, Preparation and characterization of proton (H^+) conducting solid blend polymer electrolytes based on PEO / P (VdF-HFP) incorporated with NH_4SCN. *Journal of Non-Crystalline Solids*, **579** (2022) 121368. https://doi.org/10.1016/j.jnoncrysol.2021.121368.

3. **S. Shenbagavalli,** M. Muthuvinayagam, & M. S. Revathy, Characterization of lithium-based poly (ethylene oxide)/poly (vinylidene fluoride-co-hexafluoropropylene) solid blend polymer electrolytes for energy storage applications. *Ionics*, (2022). https://doi.org/10.1007/s11581-022-04794-y.

4. **S. Shenbagavalli,** M. Muthuvinayagam, & M. S. Revathy, Enhancement of electrical and electrochemical properties of sodium bromide incorporated with poly (ethylene oxide)/poly (vinylidene fluoride-hexafluoropropylene) solid blend polymer electrolytes for electrochemical double layer capacitors. *Journal of Energy Storage*, **55** (2022) 105726. https://doi.org/10.1016/j.est.2022.105726.

5. **S. Shenbagavalli,** M. Muthuvinayagam, & M. S. Revathy, Electrical properties of Mg^{2+} ion-conducting PEO: P(VdF-HFP) based solid blend polymer electrolytes. *Polymer*, **256** (2022). https://doi.org/10.1016/j.polymer.2022.125242.